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Indian Standard
METHODS FOR ASSAYING
OF PLATINUM AND
PLATINUM ALLOYS

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHODS FOR ASSAYING OF PLATINUM AND PLATINUM ALLOYS

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Indian Standard

METHODS FOR ASSAYING OF PLATINUM AND PLATINUM ALLOYS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 15 March 1965, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 In order to determine the platinum content of various grades of platinum alloys as laid down in IS : 2275-1963* methods of determination have been prescribed in this standard. Also methods for determination of other metals commonly found associated with platinum metals in platinum alloys have been included.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publication:

HILLEBRAND (WF), LUNDELL (GEF), HOFFMAN (JI) and BRIGHT (HA). Applied inorganic analysis. 1955. Ed 2. John Wiley and Sons, Inc., New York.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard prescribes the methods for assaying of platinum in various grades of platinum as specified in IS : 2275-1963*. It also prescribes methods of determination of silver, gold, copper, iridium and rhodium which are commonly found in platinum alloys.

*Grades of platinum.

†Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1960*), shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. DETERMINATION OF PLATINUM, SILVER, GOLD AND COPPER IN PLATINUM ALLOYS BY CUPELLATION METHOD (In absence of the Platinum Group Metals)

3.1 Apparatus

3.1.1 Cupellation Furnace — consisting of a suitable muffle type furnace capable of being heated uniformly up to a temperature of 1200°C.

3.1.2 Assay Balance — of the highest precision, capable of carrying one or two grams in each pan and sensitive to 0.01 mg. The balance should be so adjusted that one division of the scale traversed by the pointer corresponds to 0.02 mg.

3.1.3 Assay Weights — should be of the highest accuracy and preferably made of platinum iridium and graduated in milligrams.

3.1.4 Cupels — Cup-like vessels made of bone-ash or magnesia.

3.1.5 Rolling-Mill — a small jeweller's rolling-mill.

3.1.6 Balling Pliers — consisting of a pair of steel pliers with smooth concave chops of suitable diameter.

3.1.7 Parting Tray — consisting of a number of small thimble-like perforated cups of platinum or silica supported in a frame of the same material.

3.1.8 Hammer and Anvil — with faces kept bright and clean.

3.1.9 Trays — various forms of trays for keeping the assay-pieces, buttons, rolled fillets, cornets, etc.

3.1.10 Tongs and Forceps — of various forms for charging cupels and handling assay-pieces, etc.

3.2 Reagents

3.2.1 Check Gold — The fineness of check gold shall be 999.9.

3.2.2 Silver — pure silver free from gold.

3.2.3 Lead Foil — lead foil of pure lead free from gold and silver.

*Specification for water, distilled quality (revised).

3.2.4 Parting Acid No. 1 — Dilute nitric acid (sp-gr 1.2) containing a trace of silver nitrate free from chloride ions.

3.2.5 Parting Acid No. 2 — Dilute nitric acid (sp-gr 1.3) containing a trace of silver nitrate free from chloride ions.

3.2.6 Parting Acid No. 3 — Concentrated sulphuric acid 90 parts by volume and water 10 parts by volume.

3.3 Procedure

3.3.1 Preliminary Assay

3.3.1.1 Weigh out accurately 0.25 g of the alloy and cupel with 5 g of lead in a magnesite crucible at about 1 100°C for about 10 minutes. If a clean rounded bead is not obtained, cupel again with another 3 g of lead. When a round button regular in shape is obtained, record its weight. Run a check assay in the same furnace with a known sample of similar composition to that of the assay.

3.3.1.2 The loss in weight of the sample after correction for the loss of silver from the check assay gives the base metal in the sample.

3.3.1.3 Put the button into a lead casing weighing 3 g and recupel at 1 100°C for 3 minutes after the addition of three times its weight of gold accurately weighed and silver equal to three times the weight of the button plus the added gold. After cooling, flatten the alloy, anneal at 850°C for 20 minutes and part for 15 minutes in boiling parting acid No. 3 till evolution of gas bubbles ceases. Repeat parting with fresh acid No. 3 for another 10 to 15 minutes to ensure complete parting. Weigh the residue after washing, drying and annealing.

3.3.1.4 The difference between the weight and that of the original cupellation button obtained in **3.3.1.1** minus the weight of gold added in **3.3.1.3** gives the weight of silver in the sample.

3.3.1.5 Cupel the residue with three times its weight of silver at 1 100°C for 3 minutes. Roll the resulting button, anneal at 850°C for 20 minutes and part for 15 minutes in parting acid No. 1. Wash, anneal and weigh. The colour of the cornet should be characteristic of pure gold and should its colour incline towards steel grey, inquart again with silver, recupel and part.

3.3.1.6 The cornet represents the original gold content after allowing for the added amount. Determine platinum by difference.

3.3.2 Final Assay

3.3.2.1 Weigh accurately 0.25 g samples of the alloy and cupel at 1 100°C with lead of the requisite quantity calculated on the copper, on a magnesite cupel. If the buttons are not satisfactory, recupel with one or two grams of lead. Run a check assay with samples of similar

composition as that of the samples. The surcharge on the check should not be more than one or two parts per 1 000. Clean the buttons and weigh. Calculate the weight of copper present after correction for loss from the surcharge.

3.3.2.2 Recupel the buttons with the addition of gold accurately weighed and silver at 1 100°C using 2 g of lead. From the preliminary assay, calculate the amount of gold and silver required to be added, on the basis that one part of platinum requires a total of ten parts of gold and the total silver required to be present is two and a half times the total weight of platinum and gold (including the added amount). Clean the buttons, hammer, anneal at 850°C for 30 minutes, roll and anneal again as before and finally make into cornets. Part the cornets for 25 minutes in boiling parting acid No. 3 and repeat the operation with fresh acid for another 10 to 15 minutes after which decant the liquid as completely as possible and again boil for 15 minutes in concentrated sulphuric acid (sp-gr 1.84). Wash, dry, anneal and weigh.

3.3.2.3 Calculate the weight of silver in the alloy from the weight (platinum + gold + added gold) and the weight of button obtained in the first cupellation (platinum + gold + silver).

3.3.2.4 Recupel the cornets as before with the addition of 2.5 times the weight of silver and using 3 g of lead. Hammer the resulting buttons. Anneal, roll and part successively in boiling parting acid No. 1 and No. 2 (sp-gr 1.2 and 1.3) for twenty minutes in each. Wash, dry, anneal and weigh. The check assays should not show a surcharge of more than one or two parts per 1 000 and the colour of the cornets should be characteristic of pure gold.

3.3.2.5 The weight of the cornet minus the weight of the added gold gives the weight of gold present in 0.25 g of the alloy sample. Calculate the weight of platinum by difference.

4. DETERMINATION OF PLATINUM, SILVER, GOLD, COPPER, IRIIDIUM, PALLADIUM AND RHODIUM IN PLATINUM ALLOYS

4.1 Reagents

4.1.1 *Aqua Regia* — solution of concentrated nitric acid (see 4.1.5) and concentrated hydrochloric acid (see 4.1.6) in the ratio of 3 : 1 (v/v).

4.1.2 *Dilute Aqua Regia* — 1 : 1 (v/v).

4.1.3 *Dilute Ammonium Hydroxide* — 1 : 1 (v/v).

4.1.4 *Concentrated Ammonium Hydroxide* — 20 percent.

4.1.5 *Concentrated Nitric Acid* — sp-gr 1.42 (conforming to IS : 264-1950*).

4.1.6 *Concentrated Hydrochloric Acid* — sp-gr 1.16 (conforming to IS : 265-1962†).

4.1.7 *Sodium Pyrosulphate* — solid.

4.1.8 *Hydrogen Sulphide* — gas.

4.1.9 *Sulphur Dioxide Solution* — saturated solution of sulphur dioxide in water.

4.1.10 *Concentrated Sulphuric Acid* — sp-gr 1.84 (conforming to IS : 266-1961‡).

4.1.11 *Oxalic Acid Solution* — saturated.

4.1.12 *Dilute Hydrochloric Acid* — 1 : 1 and 1 : 99 (v/v).

4.1.13 *Dimethylglyoxime Solution* — one percent in water (w/v).

4.1.14 *Ammonium Thiocyanate Solution* — Prepare a 2 percent solution in a solution of sulphur dioxide.

4.2 Procedure

4.2.1 Weigh out accurately in duplicate 0.5 to 2 g of the sample, rolled down to 0.08 to 0.12 mm thick and cut up into small pieces, in a 250-ml beaker. Dissolve in 25 ml of dilute aqua regia in a water bath breaking up the insoluble residue of silver chloride from time to time with a glass rod until the alloy is completely decomposed. Add 150 ml of water and digest for an hour on a hot plate. If rhodium is present, it may also be co-precipitated with iridium. Set aside to cool until the supernatant liquid is perfectly clear. Filter and wash the silver chloride with water. Preserve the filtrate.

4.2.2 Dissolve the silver chloride on the filter with hot dilute ammonium hydroxide as completely as possible. Iridium, if any, will be left on the paper as a black metallic residue. Transfer the paper to a beaker and add 20 ml of concentrated ammonium hydroxide and digest for an hour. Filter into a small beaker and wash with hot ammoniacal water. Transfer the paper to a crucible, dry, ignite, reduce in atmosphere of hydrogen and weigh as metallic iridium. Calculate the percentage of iridium in the sample.

4.2.3 Combine all the ammoniacal filtrates and washings obtained in 4.2.2, concentrate to a suitable bulk and acidify with concentrated nitric

*Specification for nitric acid.

†Specification for hydrochloric acid (revised).

‡Specification for sulphuric acid (revised).

acid. Filter off the silver chloride, wash, dry and weigh. Calculate the percentage of silver.

4.2.4 Evaporate the filtrate from **4.2.3** to dryness. Add 5 ml of concentrated hydrochloric acid and evaporate. Transfer the residue completely by means of concentrated hydrochloric acid to a crucible and again evaporate to dryness. Mix the residue with 10 g of sodium pyrosulphate and fuse at a red heat for half an hour. Cool and extract the melt with hot water. Pass hydrogen sulphide through the boiling solution for half an hour and filter off any platinum and palladium sulphide that are precipitated. Wash thoroughly in hot water, dry and ignite in a porcelain crucible. Dissolve the residue in a few millilitres of dilute aqua regia in the crucible, filter off any silica and add the solution to the filtrate preserved under **4.2.1**.

4.2.5 Evaporate the solution to dryness, take up with concentrated hydrochloric acid and again evaporate to dryness. Add 200 ml of concentrated hydrochloric acid and 50 ml of saturated solution of sulphur dioxide and re-evaporate. Add another 10 ml of the sulphur dioxide solution and set to cool. Filter the clear supernatant liquid through a filter paper containing a little pulp and wash the gold in the beaker thoroughly with hot solution of concentrated hydrochloric acid. Preserve filtrate and washings.

4.2.6 Recover any gold from the filter paper by ignition of the paper and mix with the main bulk of the gold in the beaker. Dissolve gold in the beaker in 20 ml of aqua regia, filter if necessary and evaporate to dryness. Take up with 3 ml of hydrochloric acid and again evaporate to dryness. Repeat the operation to eliminate nitric acid completely. Take up the residue with 5 ml of concentrated hydrochloric acid, 10 drops of concentrated sulphuric acid and 150 ml of water. Add 50 ml of saturated solution of oxalic acid, boil the solution for 15 minutes. Add 10 ml more of oxalic acid solution and boil for 2 minutes after which leave the vessel on a steam bath for 4 hours. Filter off the gold precipitate, wash with hot dilute hydrochloric acid and finally with hot water. Dry, ignite and weigh as gold. Calculate the percentage of gold.

4.2.7 Evaporate the filtrate and washings from **4.2.6** to dryness, add 5 ml of concentrated sulphuric acid and fume until all sulphuric acid is nearly driven off. Digest the residue with 10 ml of dilute aqua regia until a clear solution is obtained. Filter, if necessary, from any silica and mix the solution with filtrate preserved under **4.2.5**. Evaporate to dryness and take up the residue with 10 ml of dilute aqua regia until a clear solution is obtained. Dilute to 250 ml. To the cold solution add a boiling solution of dimethylglyoxime to precipitate palladium. After keeping it standing for one hour, filter off the precipitate on paper and wash well with hot water. Test filtrate for completion of precipitation and preserve.

4.2.8 Transfer the paper and the precipitate to the original beaker and dissolve the precipitate in 25 ml of dilute aqua regia on a steam heater. Filter off the paper pulp and wash with hot water, ignite and dissolve any residue in dilute aqua regia and mix with main solution. Dilute the solution and reprecipitate palladium as before. Filter on a Gooch crucible, wash with hot water, dry at 110°C and weigh as palladium as before. Combine the filtrate with that preserved under 4.2.7. Calculate the percentage of palladium.

4.2.9 Evaporate to dryness and destroy dimethylglyoxime with concentrated nitric acid on a steam heater. Digest the residue with dilute hydrochloric acid (1 : 1) and take up the dissolved salts with water. Add a saturated solution of sulphur dioxide in 20 ml portions, until after standing for 1½ hours on the steam bath, the solution still smells of sulphur dioxide. Cool, and neutralize most of the free acid by adding a solution of sodium hydroxide until a permanent precipitate just begins to form. At this point, clear the solution by adding a few drops of concentrated hydrochloric acid and then add dropwise from a burette and with constant stirring 10 ml of ammonium thiocyanate solution. Let stand for several hours, preferably overnight, filter on a tight paper lined with filter paper pulp. Dissolve the precipitate and reprecipitate, wash with ammonium sulphate solution (one percent). Combine the filtrate and washings from the two precipitations and preserve the solution.

4.2.10 Evaporate to dryness and add 10 ml of concentrated nitric acid and digest on the steam bath for one hour. Heat the residue on the hot plate with 10 ml of concentrated sulphuric acid until fumes appear. Cool and digest the residue with 30 ml of dilute hydrochloric acid (1 : 1) on a steam bath, dilute to 100 ml and boil for complete solution. Filter off any silica, wash, dry and ignite the silica. Leach the silica in dilute aqua regia and evaporate the solution to dryness and expel nitric acid completely with concentrated hydrochloric acid. Take up in dilute hydrochloric acid and mix with the main solution. Dry the residue and weigh for copper.

4.2.11 Dilute the solution to 400 ml and adjust the acidity to 5 ml of hydrochloric acid in each 100 ml volume. Heat the solution to boiling and pass a rapid stream of hydrogen sulphide gas through the nearly boiling solution. Continue passage of the gas as the solution cools somewhat. Filter the solution and wash the precipitate with dilute hydrochloric acid. Dry the filter and precipitate and ignite in a porcelain crucible. Leach the metal residue with dilute hydrochloric acid, transfer to a filter and wash thoroughly with hot water. Ignite the metal and filter again strongly in air and then in hydrogen and weigh as platinum (when rhodium is absent). Calculate the percentage of platinum.

4.2.12 When rhodium is present, determine its content described under 6 and calculate the percentage of platinum by difference.

5. DETERMINATION OF IRIIDIUM IN PLATINUM-IRIDIUM ALLOYS

5.1 Reagents

- 5.1.1 *Test Lead* — free from gold and silver.
- 5.1.2 *Dilute Nitric Acid* — 1 : 4 (v/v).
- 5.1.3 *Concentrated Hydrochloric Acid* — see 4.1.6.
- 5.1.4 *Concentrated Nitric Acid* — see 4.1.5.
- 5.1.5 *Dilute Hydrochloric Acid* — 1 : 99 (v/v).
- 5.1.6 *Hydrogen* — gas.
- 5.1.7 *Zinc* — solid.
- 5.1.8 *Potassium Pyrosulphate* — solid.
- 5.1.9 *Dilute Sulphuric Acid* — 1 : 1 (v/v).

5.2 Procedure

5.2.1 Fuse 0.25 g of the alloy in duplicate with 10 times its weight of test lead, contained in a graphite crucible, for one hour at about 1 000°C. Allow the molten metal to solidify in the crucible. Remove the cooled ingot, brush it lightly to dislodge adhering carbon, and then treat it in a covered beaker, on the steam bath, with dilute nitric acid, using 5 ml of the acid for each gram of lead. When the lead is completely disintegrated, dilute the solution with an equal volume of water, and decant through a double paper (close-texture paper with a looser-texture paper superimposed on it). Wash the residue in the beaker with hot water, and pour the washings through the filter.

5.2.2 Return the filter papers and the residue to the beaker, and add in order, 15 ml of water, 5 ml of concentrated hydrochloric acid and 0.8 ml of concentrated nitric acid for each gram or less of sample taken. Digest on the steam bath until the lighter, black portion of the residue is dissolved (usually within 1½ hours) and only a dense grey residue of metallic iridium remains.

5.2.3 Dilute the solution with an equal volume of water, and filter through a double paper, like that used for the first filtration. Examine the beaker carefully to make sure that all of the very dense residue is transferred to the filter, wiping the walls with bits of ashless paper to supplement the action of the water jet from the wash bottle. Wash the filter and residue thoroughly with hot dilute hydrochloric acid, and examine the filtrate and washings to make sure that none of the residue has passed through the paper.

5.2.4 Dry the papers, and ignite in a porcelain crucible. After the papers are burnt out, ignite iridium strongly in air. Finally, ignite briefly in hydrogen, cool in hydrogen, and weigh as metallic iridium. (The iridium so obtained is purified from the contamination of iron, if any, as described under 5.2.2).

5.2.5 Fuse the metallic iridium residue from 5.2.4 with zinc, and remove the excess zinc with concentrated hydrochloric acid. Fuse zinc-iridium alloy so formed with potassium pyrosulphate. Digest the fused mass with dilute sulphuric acid. This process will leave a residue of iridium, free from iron, but contaminated with silica. Remove silica by hydrofluorization. Calculate the percentage of iridium.

NOTE — Ruthenium if present, will separate out quantitatively along with iridium and, therefore, should be taken care of.

6. DETERMINATION OF RHODIUM IN PLATINUM-RHODIUM ALLOYS

6.1 Reagents

6.1.1 *Aqua Regia* — see 4.1.1.

6.1.2 *Concentrated Hydrochloric Acid* — see 4.1.6.

6.1.3 *Dilute Sodium Hydroxide Solution* — 10 percent (w/v).

6.1.4 *Barium Chloride* — crystals.

6.1.5 *Sodium Carbonate* — anhydrous.

6.1.6 *Sodium Chloride Solution* — 2 percent (w/v).

6.1.7 *Dilute Hydrochloric Acid* — 1 : 4 and 1 : 99 (v/v).

6.1.8 *Hydrogen Sulphide* — gas.

6.2 Procedure

6.2.1 Alloys of platinum and rhodium dissolve slowly in aqua regia provided the rhodium content does not exceed 20 percent. The sample is rolled into thin ribbon and cut up into small pieces.

6.2.2 Dissolve 0.25 g of the sample in duplicate in aqua regia on a hot plate. Replace the aqua regia from time to time until solution is complete. Evaporate to dryness and repeat evaporation twice more with concentrated hydrochloric acid. Take up with water.

6.2.3 Add dilute sodium hydroxide solution to the solution of the two metal chlorides until yellow rhodium hydroxide begins to separate. After neutralizing, adjust the volume of the solution to 100 ml per gram of the total weight of the two metals. Add a mixture of equal volumes (5 ml of

each) of solutions containing 90 g of crystallized barium chloride and 36 g of anhydrous sodium carbonate per litre respectively. After addition of the suspension, heat the solution to boiling and boil for 2 to 3 minutes. Filter off the precipitate and wash several times with hot sodium chloride solution and return the precipitate with filter paper to the original beaker. Digest with dilute hydrochloric acid (1 : 4) until solution is complete and then filter and wash with dilute hydrochloric acid (1 : 99). Neutralize again with sodium hydroxide and repeat the whole operation.

6.2.4 Dilute the final solution to 150 ml and pass a current of hydrogen sulphide through the nearly boiling solution for 40 to 45 minutes. Filter the rhodium sulphide at once and wash with water containing a little ammonium chloride and ignite in a porcelain crucible. Reduce the ignited sulphide and cool in hydrogen and weigh as metallic rhodium. Calculate the percentage of rhodium.

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